PROJECT NUMBER: 1754
PROJECT TITLE: Spectroscopic Studies of Tobacco and Smoke Components
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SOLID STATE NMR OF TOBACCO COMPONENTS

A. **Objective**: Determine the composition and structure of tobacco cell wall biopolymers (Jan Wooten).

B. **Results**: The bonding pattern of the phenyl propane groups in the cell walls from several plants (wheat, a hard wood tree, and tobacco) has been investigated by hydroponically feeding the plants either [1-^{13}C], [2-^{13}C], [3-^{13}C] ferulic acid and examining the root tissue *in situ* by ^{13}C CPMAS NMR. The ferulic acid is biosynthetically incorporated into the plant cell wall lignin which acts as a natural binder between other cell wall components. When the NMR spectra of the correspondingly labeled ^{12}C plants are subtracted from the spectra of ^{13}C enriched plants to remove the natural abundance ^{13}C signals, the signals of the various lignin substructures originating from the ferulic acid are revealed. In CPMAS NMR, before signal integration can be used to determine the relative abundance of the different substructures, it is necessary to establish that the different carbon signals have similar polarization transfer times (T_{CH}) and rotating frame relaxation times ($T_{1\rho}$). Two representative samples were chosen for this determination, a [1-^{13}C] and a [2-^{13}C] labeled plant. A set of difference spectra was obtained for 8 cross polarization contact times for each case. The relative intensity of the signals in the [2-^{13}C] labeled sample remained constant (within 10%) for contact times between 1 and 6 ms, indicating that the T_{CH} and $T_{1\rho}$ for each signal is similar. A significant variation of the relative intensity of the signals in the [1-^{13}C] labeled sample was observed over the same range of contact time; thus an appropriate adjustment must be made before signal integrations can be used to determine the relative abundance of the different substructures involving the [1-^{13}C] label. This latter result was anticipated because non-protonated carbons (the ferulic acid carboxyl group, C-1) generally cross polarize less efficiently than protonated carbons. The integrated signal intensities of the [1-, 2- and 3-^{13}C] labeled plants (difference spectra) were measured as well as for the synthetic DHP lignin. These data are currently being analyzed; however, the contact time variation experiment confirms that the relative signal intensities for the [1- and 2-^{13}C] labeled plants that were previously obtained with a 1 ms contact time are accurate within 10%.